THE EFFECT OF TEMPERATURE CYCLING TYPICAL OF LOW EARTH ORBIT SATELLITES ON THIN FILMS OF ${\rm YBa_2^{Cu_3^O}}_{7-x}$

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ABSTRACT

Thin films of YBa $_2$ Cu $_3$ O $_7$ -x (YBCO) were temperature cycled to simulate conditions on a low earth orbit satellite. In one series of tests, epitaxial and polycrystalline YBCO films were cycled in temperature up to \pm 80°C in vacuum and in nitrogen for hundreds of cycles. The room temperature resistance of an epitaxial YBCO film increased by about 10%, but the superconducting transition temperature was unchanged. The largest changes were for a polycrystalline YBCO film on oxidized silicon with a zirconia buffer layer, for which the transition temperature decreased by 3 K. An extended test was carried out for epitaxial films. After 3200 cycles (corresponding to about 230 days in space) transition temperatures and critical current densities remained unchanged.

INTRODUCTION

The refrigeration of superconductors in space poses a challenging problem. The problem could be less severe if superconducting materials were not cooled when not in use. Temperature cycling experiments of YBCO thin films were carried out in laboratory chambers to simulate temperature exposure in space missions.

Thermal cycling experiments were done to simulate a large number of eclipses of a low earth orbit satellite. Electrical measurements were performed to search for possible degradation in room temperature resistance, the zero resistance transition temperature, and the critical current density. The maximum number of cycles corresponds to more than half a year in space.

EXPERIMENTAL CONDITIONS

Thin films of YBCO were formed by coevaporation of Y, BaF_2 , and Cu and furnace annealing in wet oxygen at 850°C for 3.5 h. The substrates used in this study were (100) $SrTiO_3$ and (100) $LaAlO_3$ for epitaxial films, and polycrystalline alumina and oxidized silicon with zirconia buffer layers for polycrystalline YBCO films. Processing, electrical, and microstructural studies of these types of films have been published [1-4].

Temperature cycling in vacuum is more relevant to space conditions, but cycling is relatively slow (10 cycles per day in the chamber used). For more extensive thermal cycling, a separate chamber with a nitrogen ambient was used, which allowed a cycling rate of 60 cycles per day.

Two groups of samples were used. The first consisted of an epitaxial YBCO film on (100) SrTiO $_3$, and polycrystalline YBCO films on a zirconia buffer layer on oxidized silicon and on polycrystalline alumina. These samples were unpatterned, and subjected to a few hundred cycles, as explained below. The second set of samples were patterned by photolithography and a weak nitric acid etch to produce a bridge 400 μm long and 20 μm wide. Silver contacts were evaporated on these samples and annealed at 550°C for 0.5 h to provide low resistance contacts for resistivity and critical current measurements. All samples were characterized by four probe electrical measurements by using pressure contacts. A 1 μV criterion for the determination of the critical current density was used for the patterned samples. The patterned samples were cycled in nitrogen for thousands of cycles, as explained in more detail in the next section.

RESULTS

Results for two samples are shown in Figures 1 and 2. Each sample had a cumulative exposure. The temperature cycling stages referred to in the figures are as follows (200 cycles corresponds to about 14 days in space):

- 1. Before temperature cycling
- 2. After 5 cycles at ± 50°C in vacuum
- 3. After an additional 200 cycles at \pm 50°C in vacuum
- 4. After an additional 200 cycles at ± 60°C in nitrogen
- 5. After an additional 200 cycles at ± 80°C in nitrogen.

The zero resistance transition temperature (T) remains constant, the transition width is unchanged in Fig. 1 and increases by about one degree in Fig. 2, and the room temperature resistance normalized to that before temperature cycling (R_N) increases at first but seems to stabilize at increases of about 10% in Fig. 1 and about 20% in Fig. 2.

The largest changes were observed for a 0.9 μm thick YBCO film on oxidized silicon with a 0.5 μm zirconia buffer layer. This sample was subjected to 200 cycles at \pm 80°C in nitrogen. The transition width increased from 3.1 to 3.6 K, T decreased from 86 to 83 K, and the room temperature resistance increased by a factor of 4.

An epitaxial YBCO film on (100) LaAlO $_3$ was subjected to extensive temperature cycling (3200 cycles at \pm 80 °C in nitrogen). This sample and its control (which remained under nitrogen at room temperature) were 0.2 and 0.4 μ m thick, respectively, and they were patterned so that critical current density (J $_{\rm C}$) measurements could be performed. The T of 89 K, the J of 3-5 x 10 A cm at 77 K, and the transition width of 1-2 K were unchanged for these samples after the period of testing. The normalized value of the room temperature resistance (R $_{\rm N}$) was the only parameter which showed a change after taking

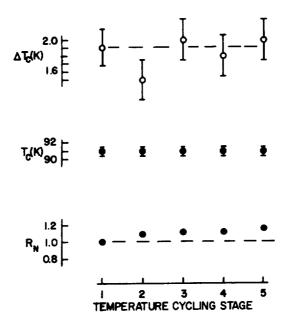


Fig. 1. Electrical parameters measured after each cycling stage (defined in the text) for a 0.5 μm thick YBCO film on (100) SrTiO3. From top to bottom: the 10 to 90% transition width, the zero resistance transition temperature, and the room temperature resistance normalized to that before temperature cycling.

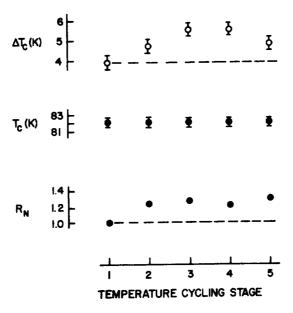


Fig. 2 Electrical parameters measured after each cycling stage (defined in the text) for a 0.7 μm thick YBCO film on polycrystalline alumina with a 0.5 μm zirconia buffer layer. The parameters are defined in the caption to Fig. 1.

into account measurement errors: an increase by 3% in the control sample and by 5% in the sample which underwent temperature cycling.

DISCUSSION

The temperature cycling experiments we have performed show that epitaxial YBCO films suffer only minor changes after temperature cycling, with somewhat greater changes found for polycrystalline YBCO films. Significant changes in electrical parameters of YBCO films have been reported after storage for a few days in nitrogen [5]. We believe that the relatively good performance of our films is related to the fact that BaF₂ was used as an evaporation source, leading to much more rugged films [6].

Even though the film annealing is performed in wet oxygen to remove the fluorine from the films, we have found by x-ray photoelectron spectroscopy that a clearly identified about 1 atomic % fluorine abundance remains in the surface region. We speculate that the fluorine improves the degradation performance of these films. As suggestive evidence we cite Tressaud et al. [7], who find for YBCO that a thin surface layer containing fluorine provides protection against hydrolysis and gas exchange, and Vasquez et al. [8], who report reduced reactivity to air for YBCO treated with HF.

CONCLUSION

The results of this study are encouraging for the use of epitaxial YBCO films in low earth orbit satellites from the viewpoint of temperature cycling, since the transition temperature and critical current density do not change after extensive temperature cycling. Thus, even with the lack of protective layers, the thin YBCO films do not necessarily have to be cooled when not in use.

REFERENCES

- 1. A. Mogro-Campero, L.G. Turner, E. L. Hall, and M.C. Burrell, "Characterization of thin films of Y-Ba-Cu-O on oxidized silicon with a zirconia buffer layer," Appl. Phys. Lett. <u>52</u> (24), 2068-2070, 1988.
- 2. A. Mogro-Campero, L.G. Turner, and G. Kendall," Thickness and annealing dependence of the superconducting transition temperature of $YBa_2Cu_3O_7$ thin films on oxidized silicon and polycrystalline alumina substrates, "Appl. Phys. Lett. $\underline{53}$ (25), 2566-2568, 1988.
- 3. A. Mogro-Campero, L.G. Turner, and E.L. Hall," Large differences of critical current density in thin films of superconducting $YBa_2^{Cu}_3^{0}_{7-x}$," J. Appl. Phys. 65 (12), 4951-4954, 1989.
- 4. A. Mogro-Campero, L.G. Turner, E.L. Hall, M.F. Garbauskas, and N. Lewis, "Epitaxial growth and critical current density of thin films of $YBa_2^{Cu}_3^{O}_{7-x}$

- on LaAlO $_3$ substrates," Appl. Phys. Lett. <u>54</u> (26), 2719-2721, 1989.
- 5. B.-Y. Tsaur, M.S. Dilorio, and A.J. Strauss, "Preparation of superconducting YBa₂Cu₃O_x thin films by oxygen annealing of multilayer metal films," Appl. Phys. Lett. 51 (11), 858-860, 1987.
- 6. P.M. Mankiewich, J.H. Scofield, W.J. Skocpol, R.E. Howard, and A.H. Dayem, "Reproducible technique for fabrication of thin films of high transition temperature superconductors," Appl. Phys. Lett. <u>51</u> (21), 1753-1755, 1987.
- 7. A. Tressaud, B. Chevalier, B. Lepine, J.M. Dance, L. Lozano, J. Grannec, J. Etourneau, R. Tournier, A. Sulpice, and P. Lejay, Passivation process of high T superconductors YBa₂Cu₃O_{7-x} by fluorine gas treatment, Modern Phys. Lett. B^c2 (10), 1183-1188, 1988.
- 8. R.P. Vasquez, B.D. Hunt, and M.C. Foote, Reduced reactivity to air on HF-treated YBa $_2^{\text{Cu}}_3^{\text{O}}_{7-x}$ surfaces, Appl. Phys. Lett. $\underline{54}$ (23), 2373-2375, 1989.